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Green and Easy Synthesis of Xanthenes Using Formic Acid as Bio-Based and Green Catalyst under Solvent-Free Conditions

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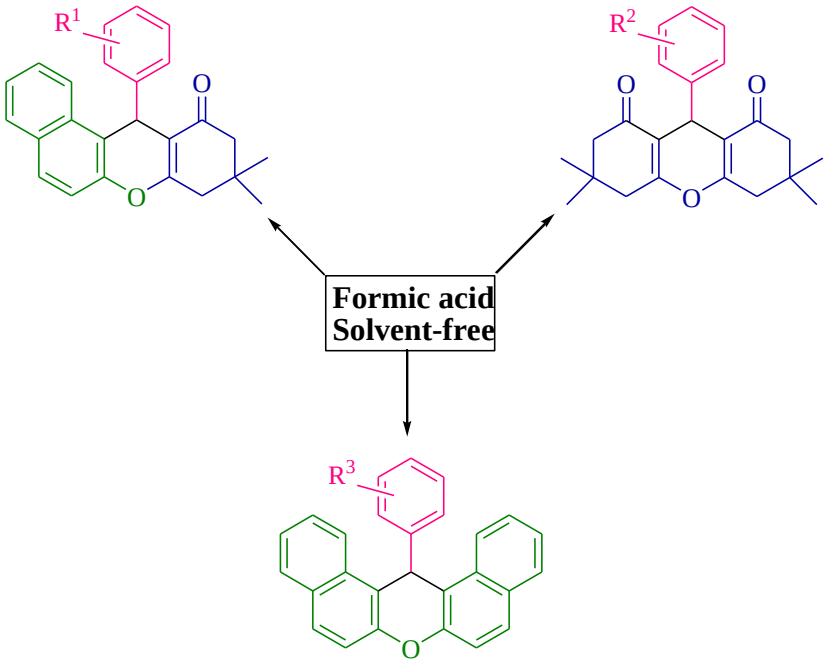
ABSTRACT

In this research study, a green synthetic route for eco-safe and solvent-free preparation of 12-aryl-tetrahydrobenzo[*α*]xanthene-11-ones, 1,8-dioxo-octahydroxanthenes and 14-aryl-14*H*-dibenzo[*α,j*]xanthenes using formic acid as a bio-based, natural and versatile catalyst was developed. The notable advantages of this green approach are use of bio-based, natural, easy-to-handle and available readily green catalyst, absence of hazardous organic solvents, solvent-free conditions with good to high yields and short reaction times and one-pot reactions. Furthermore, one of the source of environmental pollutions is the usage of organic solvents under reflux conditions and the need for column chromatography to purity the products. In this present work, the products were obtained through simple filter with no need column chromatographic separation.

GRAPHICAL ABSTRACT



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Introduction

Due to the widespread applicability of heterocyclic compounds in nature and life, their synthesis has been highly regarded. An important feature of xanthenes compounds is their anti-plasma [1] and anti-inflammatory [2] properties. Moreover, due to the fact that these fluorescent materials are pH-sensitive, they are used to visualize biomolecules [3, 4], laser technology [5, 6], luminescent dyes [7], and sensitizers in photodynamic therapy [8].

In recent decades, some methods have been established to use various catalysts including, citric acid [9], salicylic acid [10], ascorbic acid [11], Fe_3O_4@SiO_2-SO_3H [12], $NaHSO_4/SiO_2$ [13], NO_2–FePc/C [14], sulfamic acid [15], DSIMHS [16], ceric ammonium nitrate [17], trityl chloride [18], silica sulfuric acid [19], $Sr(OTf)_2$ [20], [cmmim][BF_4] [21], zinc oxide nanoparticle [22], [Hbim]BF_4 [23], [BMim][BF_4] [24], SFP [25], SiO_2-Pr-SO_3H [26], nano-alumina sulfuric acid [27],[H-NMP][HSO_4] [28] diatomite-SO_3H [29], [PSMIM]Cl [30], boric acid [31], 2,6-pyridinedicarboxylic acid [32], γ-Fe_2O_3–HAp-Fe^{2+}NPs [33] and 2-(sulfooxy)ethyl]sulfamic acid [34] to prepare these compounds. The limitations of some of these methods are difficult work-up, low yield, expensive and toxic catalyst, longer reaction time, high temperature and the use of highly acidic conditions. In order to develop safe, highly efficient, and environmentally friendly methods as a section of our continuing research program as part of our research program that is still in progress [35-44]. In this work we developed a green and easy one-pot method, using formic acid as a bio-basic and green catalyst [45] under the thermal and solvent-free situations. This method can be a great alternative to the previous methods due to the use of green, readily, low-cost, and highly efficient catalyst, simple work-up, short reaction time and good to high yield.

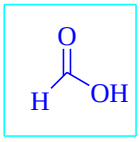


Figure 1. Structure of formic acid

Materials and Methods

General

An Electro thermal 9100 apparatus was utilized to determine the melting point of all compounds. Bruker DRX-400 Avance instruments are also used to record the nuclear magnetic resonance, ^{1}HNMR spectra using $CDCl_3$ as a solvent. All the reagents and solvents used in this research were purchased from the Merck, Fluka, and Acros and used without further treatment.

Generic approach

4a-k: A mixture of **1**, **2a-k**, **3** and formic acid (10 mol%) by heating for convenient time. The mixture is cooled to room temperature after the reaction is completed and then ethanol adds to it. The resulting precipitates are then separated using a filter and re-crystallized in order to obtain pure products from ethanol (**4a-k**).

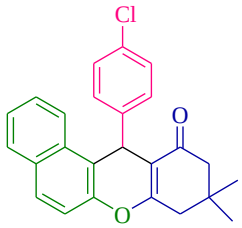
5a-j: A mixture of **3**, **2a-j**, and formic acid (10 mol%) by heating for convenient time. The mixture is cooled to room temperature after the reaction is completed and then ethanol adds to it. The resulting precipitates are then separated using a filter and re-crystallized in order to obtain pure products from ethanol (**5a- j**).

6a-j: A mixture of **1**, **2a-j** and formic acid (10 mol%) by heating for convenient time. The mixture is cooled to room temperature after the reaction is completed and then ethanol adds to it. The resulting precipitates are then separated using a filter and re-crystallized in order to obtain pure products from ethanol (**6a-j**).

Melting points and ^{1}HNMR were used to characterize the products.

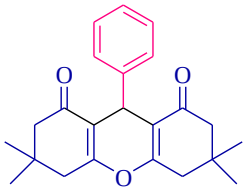
The data for the spectra of chosen and known products are given below:

Table 2, entry 3: Yield: 76%; M.p. 174-176 °C.



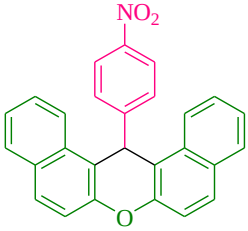
^{1}HNMR (400 MHz, $CDCl_3$): 0.98 (3H, s, CH_3), 1.15 (3H, s, CH_3), 2.24 (2H, dd, *J*= 16 Hz, CH_2), 2.53 (2H, s, CH_2), 5.62 (1H, s, CHAr), 7.36-7.91 (10H, m, ArH).

Table 4, entry 3: Yield: 88%; M.p. 204-206 °C.



^{1}H NMR (400 MHz, $CDCl_3$): 1.03 (6H, s, $2CH_3$), 1.10 (6H, s, $2CH_3$), 2.25 (4H, dd, *J*= 16 Hz, $2CH_2$), 2.42 (4H, s, $2CH_2$), 4.76 (1H, s, CH), 7.17-7.26 (5H, m, ArH).

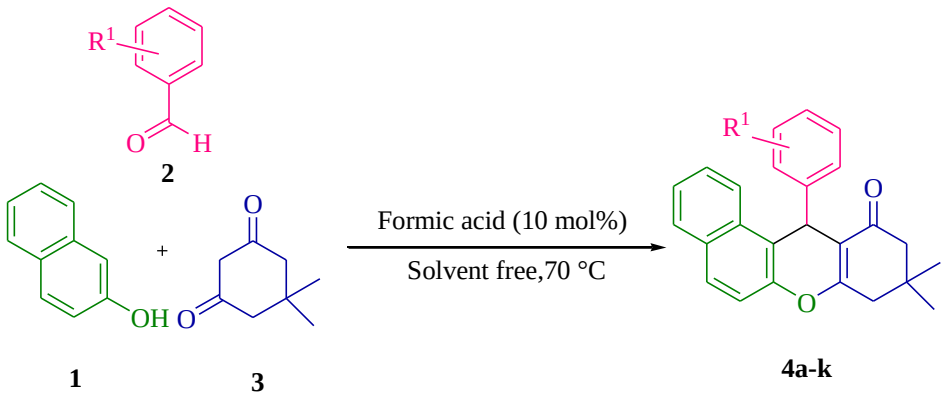
Table 6, entry 1: Yield: 89%; M.p. 306-307 °C.



^{1}H NMR (400 MHz, $CDCl_3$): 6.56 (1H, s, CH), 7.28 (2H, d, *J*=8.4 Hz, ArH), 7.36 (2H, d, *J*=8.4 Hz, ArH), 7.58 (2H, t, *J*=7.6 Hz, ArH), 7.66 (2H, d, *J*=8.6 Hz, ArH), 7.87 (4H, t, *J*=7.6 Hz, ArH), 7.96 (2H, d, *J*=8.4 Hz, ArH), 8.24 (2H, d, *J*=8.6 Hz, ArH).

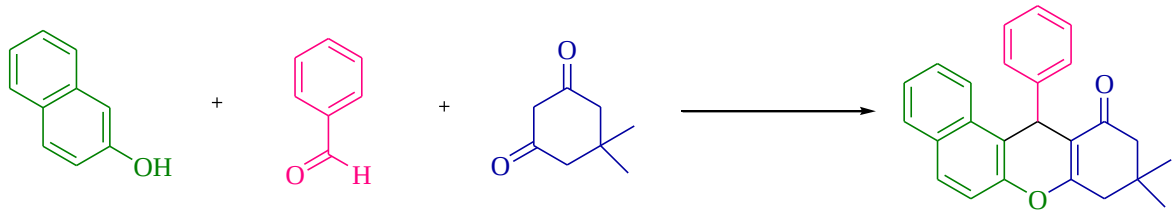
Results and Discussion

First, to optimize the reaction, preparation of 4 g under typical thermal condition. Afterward, the reaction was tested in the presence of 10 mol% formic acid at a room temperature of 80 °C without the use of solvent (Table 1). As seen in Table 1, when the reaction takes place at a temperature of 70 °C, the increase in reaction temperature to 80 °C had not a significant effect on improving reaction results (Table 1, entry 8). In another study, compound 4gwere tested using various molar ratios of formic acid at 70 °C under solvent-free conditions (Table 1). According to Table 1, 10 mol% of the catalyst are adequate for the reaction to proceed efficiently at 70 °C (Table 1, entry 3). The generality and efficiency of the method were investigated with arylaldehydes bearing electron withdrawing substituents, electron-donating substituents (**2**, 1.0 mmol), following the optimization of reaction conditions (Scheme 1). Table 2 demonstrates that the method used is a general and efficient method, and all reactions were performed with good to high yield and in a relatively short time.



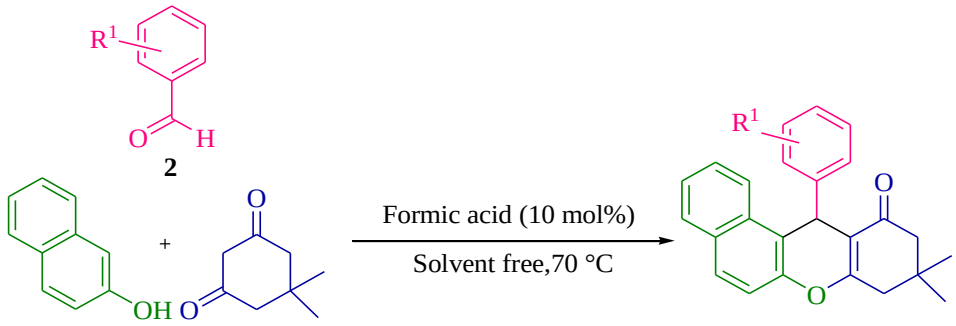
Scheme 1. Synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones.

Table 1. Optimization results of reaction conditions for synthesis of **4g**.



Entry	Formic acid (mol %)	Temperature (°C)	Time (min)	Isolated Yields (%)
1	Catalyst free	70	360	trace
2	5	70	40	39
3	**10**	**70**	**20**	**86**
4	10	rt	360	trace
5	10	40	75	31
6	10	50	50	46
7	10	60	30	68
8	10	80	20	85
9	15	70	20	86

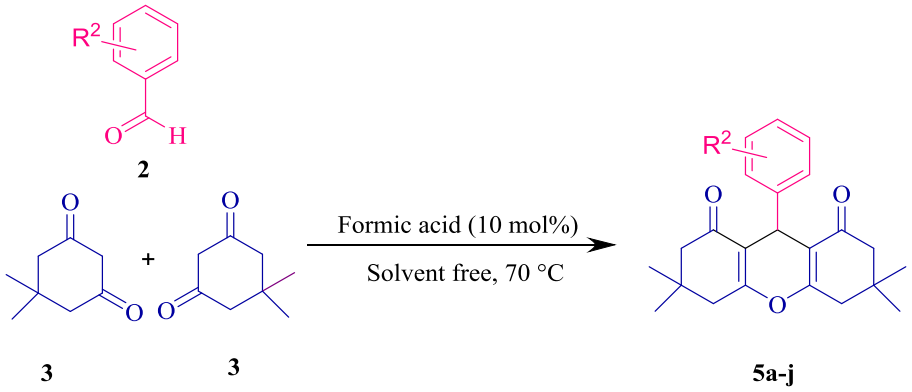
Table 2. Synthesis of **4a-k**.



Product	R^1	Time (min)	Isolated Yields (%)	M.p.°C	M.p.°C
4a	4-F	15	90	185-187	184-185 [12]
4b	3-Br	35	78	162-164	161-164 [18]
4C	4-Cl	30	76	174-176	176-178 [16]
4d	4-OH	35	80	223-225	222-223 [16]
4e	3-Me	20	89	178-180	178-180 [15]
4f	3-NO_2	15	88	166-168	167-169 [12]
4g	H	20	86	150-152	148-150 [16]
4h	3-OMe	25	85	203-205	202-204 [16]
4i	4-NO_2	15	91	175-177	175-178 [18]
4j	4-Br	35	75	182-184	184-186 [16]
4k	4-Me	20	86	170-172	171-173 [16]

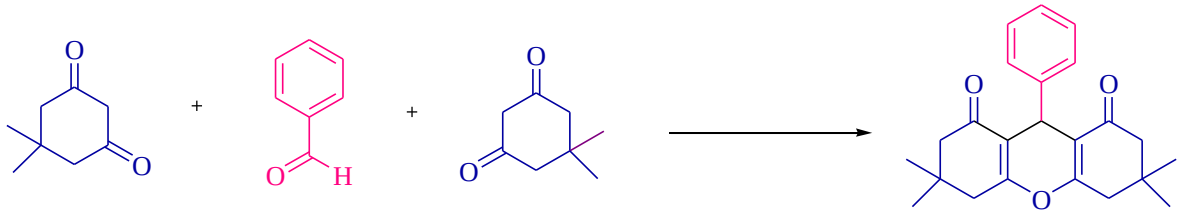
Then, all our attention was focused on the synthesis of 5a-j. In this case, the reaction of benzaldehyde (1.0 mmol) and dimedone (2.0 mmol), in the absence of solvent, was selected as the model for optimization reaction conditions. According to many studies on the model reaction, we concluded that when the mol% of the formic acid is less than 10%, the yield of the products is

low and more time is needed to produce them. In contrast, if more than 10 mol% of formic acid is used, the yield does not improve and the reaction takes same time to complete (Table 3, entries 9). Based on this result, it became clear that the extra catalyst does not increase the reaction rate, so in all reactions 10 mol% of formic acid is used as the catalyst. The effect of different temperatures on the model reaction was investigated using 10 mol% of formic acid and without the presence of a solvent, and the finest outcomes were achieved when the temperature was considered to be 70 °C (Table 3, entry 3). According to Table 3, it is observed that with increasing temperature up to 80 °C, the production yield increases, but if the temperature increases further, the yield will not be higher than it is at 70 °C (Table 3, entry 8). According to the results in Table 4 and Scheme 2, the reactions of diverse aromatic aldehydes with dimedone were carried out effectively, by producing desirable products, good to high yields, and in a relatively short time.



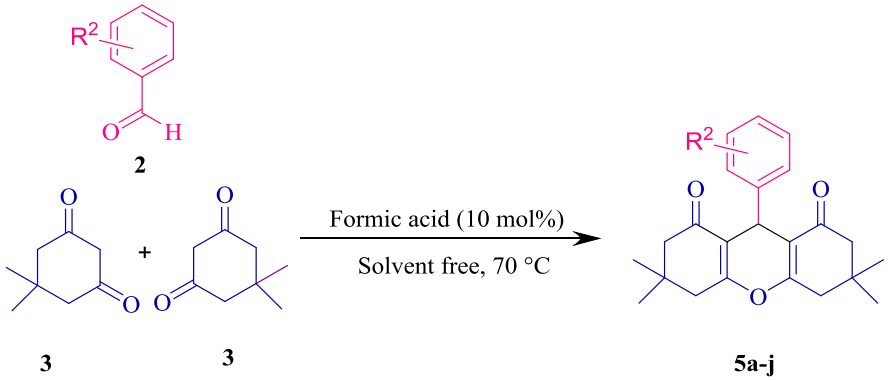
Scheme 2. Synthesis of 1,8-dioxo-octahydroxanthenes.

Table 3. Optimization results of reaction conditions for synthesis of **5c.**



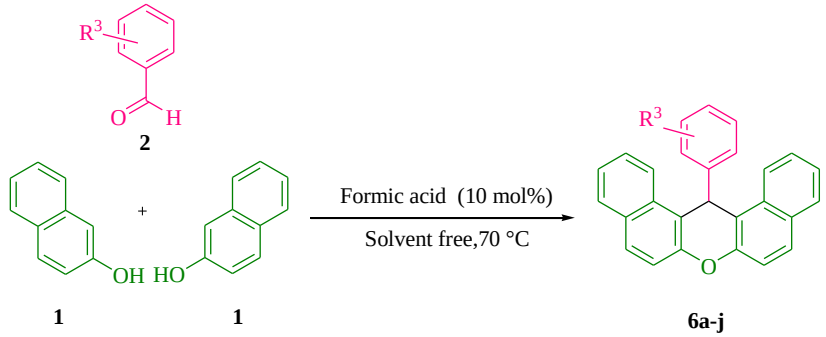
Entry	Formic acid (mol %)	Temperature (°C)	Time (min)	Isolated Yields (%)
1	Catalyst free	70	360	trace
2	5	70	20	57
3	**10**	**70**	**10**	**88**
4	10	rt	360	trace
5	10	40	65	29
6	10	50	45	50
7	10	60	20	65
8	10	80	10	88
9	15	70	10	89

Table 4. Synthesis of **5a-j**.



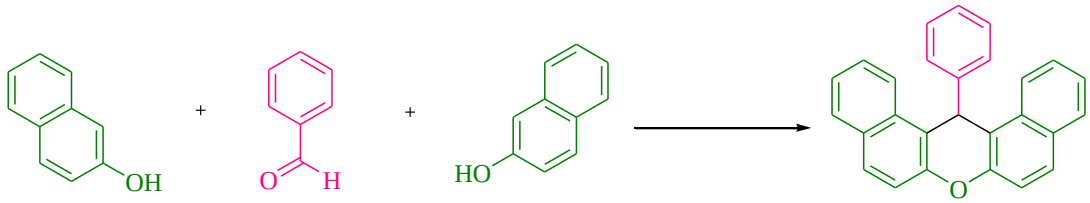
Product	R^2	Time (min)	Isolated Yields (%)	M.p.°C	M.p.°C
5a	3-Br	20	79	191-193	192-194 [21]
5b	3,4,5-OMe	20	86	187-189	186-188 [21]
5c	H	10	88	204-206	206-208 [16]
5d	5-F	10	90	193-195	193-195 [16]
5e	4-NO_2	10	89	224-226	222-224 [21]
5f	4-OH	20	76	245-247	246-248 [21]
5g	3-NO_2	10	87	169-171	171-172 [12]
5h	4-OMe	15	84	243-245	241-243 [12]
5i	4-Me	10	86	214-216	216-218 [21]
5j	4-Cl	25	82	237-239	235-238 [12]

The aim of this study was to report the application of this reagent for the synthesis of 6a-j in solvent-free conditions (Scheme 3). The condensation of **1**and **2e** and formic acid (10 mol%) was performed by heating to a temperature of 70 °C and without the presence of solvent to optimize the reaction, which was completed after 20 min (Table 5, entry 3). According to Table 5, it can be concluded that increasing the temperature and amount of acid has no effect on the reaction result (Table 5, entry 8, 9). To demonstrate the role of formic acid as a catalyst and its effectiveness in the reaction, the preparation 14-phenyl-14*H*-dibenzo[*α,j*]xanthenes was performed without the use of a catalyst, and after 360 minutes, a trace of the product was obtained (Table 5, entry 1). Therefore, it is clear that the use of formic acid to perform reaction is necessary (Table 5). After optimizing the reaction conditions, different aryl aldehydes were considered for the condensation reaction with *β*-naphthol, under the selected conditions. The results are illustrated in Table 6.



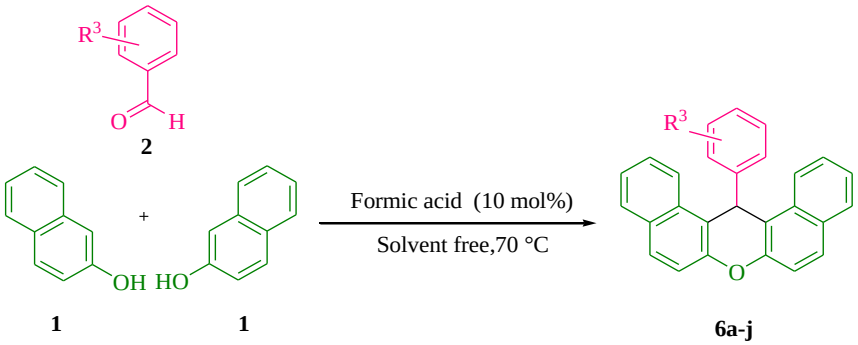
Scheme 3. 14-aryl-14*H*-dibenzo[*α,j*]xanthenes.

Table 5. Optimization results of reaction conditions for synthesis of **6e**



Entry	Formic acid (mol %)	Temperature (°C)	Time (min)	Isolated Yields (%)
1	Catalyst free	70	360	trace
2	5	70	35	53
3	**10**	**70**	**20**	**85**
4	10	rt	360	trace
5	10	40	70	26
6	10	50	60	47
7	10	60	35	64
8	10	80	20	87
9	15	70	20	86

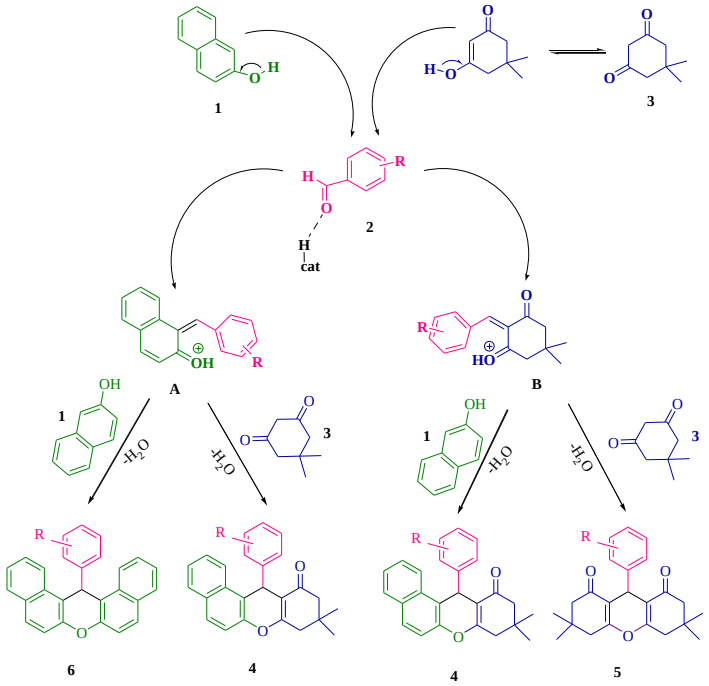
Table 6. Synthesis of **6a-j**.



Product	R^3	Time (min)	Isolated Yields (%)	M.p.˚C	M.p.˚C
6a	4-NO_2	15	89	306-307	308-309 [28]
6b	4-OMe	20	81	204-206	204-205 [16]
6c	3-Br	30	76	191-193	191-193 [12]
6d	4-F	10	89	241-243	240-242 [22]
6e	H	20	85	184-186	183-184 [29]
6f	4-Cl	30	78	290-292	289-290 [29]
6g	4-Me	15	86	225-227	227-228 [16]
6h	3-Me	10	89	197-199	197-198 [28]
6i	4-Br	30	78	295-297	297-298 [16]
6j	3-NO_2	15	86	213-215	212-213 [28]

Suggested mechanistic paths in the presence of formic acid are depicted in scheme 4. According to this mechanism, formic acid activates the aldehyde by donating the proton to the oxygen atom of aldehyde. After that nucleophilic(**1**) or (**3**) attacks the carbonyl group of the activated aldehyde and produces products A and B by removing H_2O. The addition of intermediates to 1 or 3 has led to the creation of an adduct circular intermediate, which, with the assistance of two hydroxyl groups to produce xanthenes, undertakes an intermolecular cyclization (Scheme 4).

Comparison of catalytic ability some of catalysts reported in the literature for synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one (**4**), 1,8-dioxo-octahydroxanthenes (**5**) and 14-aryl-14*H*-dibenzo[α,j]xanthenes (**6**) are shown in Table 7, 8, 9.



Scheme 4. Suggested mechanistic paths

Table 7. Comparison of catalytic ability some of catalysts reported in the literature for synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives [a]

Entry	Catalyst	Conditions	Time/Yield (%)	References
1	Fe_3O_4@SiO_2-SO_3H	solvent-free, 110 °C	30 min/95	[12]
2	$NaHSO_4/SiO_2$	CH_2Cl_2, Reflux	300 min/91	[13]
3	NO_2–FePc/C	EtOH, Reflux	30 min/91%	[14]
4	DSIMHS	solvent-free, 55 °C	20 min/93	[16]
5	CAN	microwave irradiation, 120 °C	120 min/85	[17]
6	$Sr(OTf)_2$	1,2-dichloroethane, 80 °C	300 min/85	[20]
7	**formic acid**	**solvent-free, 70 °C**	**20 min/86**	**This work**

[a] Based on the three-component reaction of β-naphthol (1.0 mmol); benzaldehyde (1.0 mmol) and dimedone (1.0 mmol)

Table 8. Comparison of catalytic ability some of catalysts reported in the literature for synthesis of 1,8-dioxo-octahydroxanthene derivatives [a]

Entry	Catalyst	Conditions	Time/Yield (%)	References
1	Fe_3O_4@SiO_2-SO_3H	solvent-free, 110 °C	4 min/94	[12]
2	DSIMHS	solvent-free, 55 °C	4 min/95	[16]
3	[cmmim][BF_4]	microwave irradiation	2 min/92	[21]
4	[Hbim]BF_4	microwave irradiation	45 min/85	[23]
5	[BMim][BF_4]	$Mg(BF_4)_2$, 80 °C	30 min/97	[24]
6	**formic acid**	**solvent-free, 70 °C**	**10 min/88**	**This work**

[a] Based on the three-component reaction of dimedone (2.0 mmol) and benzaldehyde (1.0 mmol)

Table 9. Comparison of catalytic ability some of catalysts reported in the literature for synthesis of 14-aryl-14*H*-dibenzo[*α,j*]xanthenes derivatives [a]

Entry	Catalyst	Conditions	Time/Yield (%)	References
1	Fe_3O_4@SiO_2-SO_3H	solvent-free, 110 °C	30 min/94	[12]
2	DSIMHS	solvent-free, 90 °C	3 min/94	[16]
3	[BMim][BF_4]	$Mg(BF_4)_2$, 80 °C	15 min/95	[24]
4	SFP	solvent-free, 90 °C	30 min/98	[25]
5	SiO_2-Pr-SO_3H	solvent-free, 125 °C	20 min/98	[26]
6	[H-NMP][HSO_4]	solvent-free, 110°C	12 min/94	[28]
7	diatomite-SO_3H	solvent-free, 90°C	10 min/93	[29]
8	**formic acid**	**solvent-free, 70°C**	**20 min/85**	**This work**

[a] Based on the three-component reaction of *β*-naphthol (2.0 mmol) and benzaldehyde (1.0 mmol).

Conclusion

In this work, we demonstrated that a bio-based, natural and efficient catalyst, formic acid, can be used in the eco-safe, one-pot and facile synthesis of 12-aryl-tetrahydrobenzo[*α*]xanthene-11-ones, 1,8-dioxo-octahydroxanthenes and 14-aryl-14*H*-dibenzo[*α,j*]xanthenes under thermal and solvent-free conditions with good to high yields and short reaction times through simple filter with no necessity of chromatographic purification steps. Use of the inexpensive and easy to handle formic acid as a bio-based, versatile and natural catalyst, straightforward work-up, simplicity of operation, absence of hazardous organic solvents, eco-safe and one-pot procedure are among the other added advantages that make this approach an attractive alternative for the synthesis of these biologically active compounds.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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